
वायु प्रदूषण को मापने की पद्धति
भाग 25 अमोनिया

**Methods for Measurement
of Air Pollution**
Part 25 Ammonia

ICS 13.040.20

© BIS 2018



भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

FOREWORD

This Indian Standard (Part 25) was adopted by the Bureau of Indian Standards, after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.

The undesired impacts of pollutants on flora and fauna is of high concern to scientists and technologists, in particular, and to common people, in general. Ammonia is one such pollutant. Gaseous ammonia (NH_3) is the most abundant alkaline gas in the atmosphere. The largest source of ammonia (NH_3) emissions is agriculture, including animal husbandry and ammonia (NH_3) based fertilizer applications. Other sources of ammonia (NH_3) include industrial processes, vehicular emissions and volatilization from soils and oceans.

Atmospheric ammonia presents three major environmental issues:

- a) Odor — Ammonia has a strong and unpleasant odor.
- b) Ammonia at high levels is toxic to humans and animals. It is known to cause irritation to skin, eyes and respiratory tract.
- c) Ammonia reacts with atmospherically formed sulfuric and nitric acids and forms precipitated salts which contribute to ambient level of fine particles.

Keeping the above in mind, proper methodology to accurately determine the concentration of ammonia in ambient atmosphere is of utmost importance. This standard describes Indophenol method for the measurement of ammonia in ambient air.

In reporting the results of a test or analysis in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 25 AMMONIA

1 SCOPE

This standard prescribes the Indophenol method for measurement of ammonia in ambient air.

2 REFERENCE

‘The standards given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.’

<i>IS No.</i>	<i>Title</i>
265 : 1993	Hydrochloric Acid
266 : 1993	Sulphuric Acid
376 : 1986	Sodium Hydroxide, Analytical Reagent
538 : 2000	Phenol (Carbolic Acid)
1070 : 1992	Reagent Grade Water
4167 : 1980	Glossary of terms relating to air pollution (<i>first revision</i>)
5182 (Part 2) : 2001	Methods for measurement of air pollution: Part 2 Sulphur dioxide (<i>first revision</i>)

3 TERMINOLOGY

For the purpose of this standard the definitions given in IS 4167 shall apply.

4 PRINCIPLE

Ammonia in the atmosphere is collected by bubbling a measured volume of air through a dilute solution of sulfuric acid to form ammonium sulfate. The ammonium sulfate formed in the sample is analyzed colorimetrically by reaction with phenol and alkaline sodium hypochlorite to produce indophenol, a blue dye. The reaction is accelerated by the addition of sodium nitroprusside as catalyst.

5 RANGE AND SENSITIVITY

5.1 With a sampling rate of 1 to 2 l/min, a concentration range of 20 to 700 µg/m³ (0.025 to 1 ppm) in air may be determined with a sampling time of 1 h.

5.2 The lower limit of detection of the analysis is 0.2 µg NH₃/ml.

6 INTERFERENCES

6.1 Prefilters may remove some gaseous ammonia (*see* clause 10.1.1).

6.2 Ferrous, chromous, and manganous ions if present in mg amounts cause positive interference in the analytical procedure because of precipitation. Copper ions inhibit color development strongly and therefore cause negative interference. Addition of Ethylene Diamine Tetraacetic Acid (EDTA) prevents these effects. Nitrite and sulfite interfere if present in 100-fold excess. Based on tests with solutions, formaldehyde causes a negative interference of 10 to 15 percent.

6.3 Interfering particulate matter in the air can be removed by filtration of the air sample.

7 PRECISION AND ACCURACY

7.1 Replicate samples, collected with glass prefilters and analyzed manually show a relative coefficient of variation of 30 percent in the 0.7 to 21 µg/m³ (1 to 30 ppb) range. This coefficient varies with concentration of atmospheric ammonia and decreases to 5 percent in the 700 µg/m³ (1 ppm) range.

7.2 No accuracy data are available.

8 APPARATUS**8.1 Vacuum Pump**

Any vacuum pump that can maintain a vacuum of 60 kPa with an air flow of 5 l/min.

8.2 Flow Measuring Device

A calibrated flow meter or a critical orifice to measure or control the air flow from 1 to 2 l/min. The flow meter should be calibrated under conditions of use.

8.3 Absorber

A fritted bubbler, midjet impinger, or other gas scrubber designed for a flow rate of 1 to 2 l/min with 10 ml or more absorbing solution is suitable for sampling.

8.4 Prefilter Holder

An open-face polytetrafluoroethylene (PTFE), polycarbonate thermoplastic, or similar filter holder, which can be connected in line before the bubbler, may be used as per objective of monitoring.

8.5 Prefilters

Organic free glass fibre filter used for air sampling for suspended particulate matter are satisfactory.

8.6 Spectrophotometer

Capable of measuring the absorbance at 630 nm, with wavelength accuracy of 2 nm or less.

8.7 Glassware

Low-actinic glassware or vessels must be used for analysis. The glassware must be rinsed with 1.2 N hydrochloric acid (HCl) and washed at least 6 times with reagent water before immediate use.

9 REAGENTS

9.1 Purity

All chemicals should be of analytical reagent grade.

9.2 Water

Water (Grade 1) must meet specification given in IS 1070, and shall be ammonia free.

9.3 Absorbing Solution

Dilute 3.0 ml of concentrated sulphuric acid (H_2SO_4) (18 M) (*see* IS 266) to 1 l with water to obtain 0.1 N sulphuric acid (H_2SO_4).

9.4 Sodium Nitroprusside [Sodium Nitrosylpentacyanoferrate (III)]

Dissolve 2 g sodium nitroprusside in 100 ml of water. The solution keeps well in the refrigerator for 2 months.

9.5 Sodium Hydroxide, 6.75 M

Dissolve 270 g sodium hydroxide (*see* IS 376) in about 1 l of distilled water. Boil down to 600 ml in order to volatilize the ammonia contained in the reagent. Cool and make up the volume to 1 l using ammonia free reagent water (9.2). Store in polyethylene bottle.

CAUTION: This solution is extremely caustic. Prevent contact with skin or eyes.

9.6 Sodium Hypochlorite Solution (0.1 N)

Dilute 5 to 6 percent analytical reagent sodium hypochlorite with distilled water to give a 0.1 N solution (3.7 percent). The solution keeps well for 2 months in a refrigerator.

9.6.1 Standardization of Sodium Hypochlorite Solution

9.6.1.1 Acidic iodide solution — Dissolve 7 g potassium iodide (KI) in 20 ml glacial acetic acid and make up the volume to 1 000 ml with distilled water.

9.6.0.1 Standard sodium thiosulphate solution (0.1 N) — *see* 7.8 in IS 5182 (Part 2) : 2001.

9.6.0.2 Take 50 ml acidic iodide solution in a conical flask. Add 1 ml sodium hypochlorite solution. Titrate against 0.1 N sodium thiosulphate solution (**9.6.1.2**) till yellow colour appears. Add starch solution and titrate till colourless. Note the burette reading.

9.6.0.3 Calculation

Normality of NaOCl = $(\text{BR} \times \text{Normality of Na}_2\text{S}_2\text{O}_3) / \text{volume of NaOCl in ml}$

where,

BR means burette reading.

9.7 Phenol Solution 45 Percent (v/v)

Melt phenol (Grade A, *see* IS 538) by immersing a bottle containing the material in a water bath at 60°C. Pour 45 ml (50 g) in to a 100 ml warmed cylinder and fill to mark with methanol.

9.7.1 Buffer

Dissolve 50 g of sodium phosphate dodecahydrate ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$) and 74 ml of 6.75 M sodium hydroxide (NaOH) in distilled water and make up the volume to 1 l with distilled water.

9.7.2 Working Hypochlorite Solution

Mix 30 ml of 0.1 N sodium hypochlorite and 30 ml of 6.75 M Sodium hydroxide and dilute to 100 ml with distilled water. Prepare fresh daily.

9.7.3 Working Phenol Solution

Mix 20 ml of the 45 percent Phenol solution with 1 ml of 2 percent Sodium nitroprusside and dilute to 100 ml with distilled water. Prepare fresh every 4 hour.

9.8 Ammonia Standard Solution

9.8.1 Ammonia stock Solution

Dissolve 3.18 g of ammonium chloride (NH_4Cl) or 3.88 g of ammonium sulphate [$(\text{NH}_4)_2\text{SO}_4$] in 1 l of distilled water (1 ml equal to 1 mg ammonia (NH_3)). Add a drop of chloroform (CHCl_3) for better preservation. The solution is stable for 2 months.

9.8.2 Ammonia Working Solution

Dilute 10 ml of the stock solution to 1 l with absorbing solution in a volumetric flask [1 ml equal to 10 µg ammonia (NH_3)]. Prepare daily.

9.8.3 Glass Cleaning Solution

Dilute 10 ml of concentrated hydrochloric acid (HCl) (12 M) (see IS 265) to 100 ml with distilled water (molarity approximately 1.2 M).

10 PROCEDURE

10.1 Sample Collection

Place 10 ml of absorbing solution in each bubbler for samples and field blanks. Cap bubbler for transport. Assemble (in order given) prefilter and holder, flow meter, bubbler and pump. Sample is collected at the rate of 1 – 2 l/min for a period of time. The period of sampling should not exceed 1 hour. Record sampling time and flow rate. After sample collection, recap the bubbler.

10.1.1 If prefilters are not used, the method will determine both gaseous ammonia and ammonium contained in particulates. At high humidity, acid gas will promote reaction on the filter causing loss of ammonia gas from the sample. In the absence of acid gases, ammonia collected momentarily on the filter during high humidity will be stripped off during sampling with little loss.

10.2 Transfer contents from bubbler to a 25 ml capacity volumetric flask (blowout residual sample from the frits if they are used). Maintain all solutions and sample at 25°C. Add 2 ml buffer and 5 ml of working phenol solution to the flask, mix and fill to about 22 ml. Then add 2.5 ml of the working hypochlorite solution and mix rapidly. Dilute to 25 ml, mix, and store in the dark at 25°C for 30 min to develop color. Measure the absorbance of the solution against a reagent blank at 630 nm, using 1 cm cells.

10.3 Field Blanks

At least one bubbler of collecting solution is carried into the field and treated in the same manner as the actual samples except that no air is drawn into the bubbler. It is treated in analysis as if it was a sample. The use of the reagent blank is to determine whether sampling glassware is causing any appreciable contamination.

11 CALIBRATION AND STANDARDIZATION

11.1 Preparation of standards

Pipet required aliquot of the working standards solution into 25 ml glass stoppered volumetric flask and make up the volume to 10 ml with absorbing solution to get 5 point calibration in the range of 1 – 20 µg of ammonia. Add reagents to each flask as in the procedure for analysis. Read the absorbance of each standard against the reagent blank.

11.2 Standard Curve

Plot the absorbance as the ordinate versus the concentration as abscissa on linear graph paper. Alternatively, determine the slope by the method of least squares.

12 CALCULATION

$$C (\mu\text{g}/\text{m}^3 \text{ NH}_3) = \frac{W}{V_0}$$

where,

C = concentration of ammonia in µg/m³,

W = µg NH₃ in 25 ml from standard curve, and

V_0 = volume of air sample in m³.

If values need to be reported after normalization, calculate volume using the following equation at 25°C and 101.3KPa (760 mm Hg):

$$V_0 = \left(\frac{F}{1000} \right) \times \left(\frac{P_s}{101.3} \right) \times \left(\frac{298}{273 + T_s} \right)$$

where,

F = Flow rate (l/min),

P_s = atmospheric pressure in kPa at sampling point, and

T_s = ambient temperature, °C at sampling point.

13 EFFECT OF STORAGE

It has been observed that if the collected liquid samples are stored for 2 days, precision of the method is lowered significantly.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 2016* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: CHD 35 (11266).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

Telephones

Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	{ 2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	{ 265 0206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	{ 2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	{ 2832 9295, 2832 7858 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE.
DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI.
HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW.
NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi